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The Influence of Alpha2 on Properties and Microstructure in Ti6Al4V

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Synopsis

Samples of Hot Isostatically Pressed (HIPped) powder and of forged bar stock Ti6Al4V which were slowly cooled from above the beta transus and subsequently held at 500°C for times up to 5 weeks, have been analysed using scanning and transmission electron microscopy and atom probe analysis. It has been shown that in the samples heat treated for 5 weeks at 500°C there is a high density of alpha2 domains, which are typically 5nm in size and that a smaller density was present in the slowly cooled samples. The fatigue and tensile properties of samples heat treated for 5 weeks at 500°C have been compared with those of samples slowly cooled from just above the transus and although no significant difference was found between the fatigue properties, the tensile strength of the heat treated samples was 5% higher than that of slowly cooled samples. The ductility of the forged samples did not decrease after heat treating at 500°C despite the strength increase. Transmission electron microscopy has been used to assess the nature of dislocations generated during tensile and fatigue deformation and it has been found that not only is planar slip observed, but dislocation pairs are not uncommon in samples aged at 500°C and some are seen in slowly cooled Ti6Al4V.

1.Introduction

The alloy Ti6Al4V is commonly referred to as the workhorse Ti alloy because it is the most widely used Ti alloy. It was developed over 50 years ago and a great deal of research has been carried out aimed at understanding how to optimise the microstructure for specific properties, but despite that there are some areas of uncertainty and this paper is focussed mainly on just one aspect – the development of order in the alpha phase and its influence on properties. Earlier experimental work [1, 2] has shown that the alpha phase orders; this ordering is expected from phase diagram modelling [3]. Other work on deformed Ti6Al [2] had shown that weak fringes were observed in TEM images within the planar slip bands and these were interpreted as being due to the destruction of short range order by the dislocations. In fact there have been many examples [e.g refs 5 -9] of the observation of planar slip in Ti alloys with more than 4% Al and of fringes in other alloys which could well have been due to the destruction of order. The work presented in this paper has been carried out both to define the nature of the ordered alpha2 and the influence of this alpha 2 on the tensile and fatigue properties.

2. Experimental

The Ti6Al4V used in most of this work was prepared from PREP powder that was HIPped (Hot Isostatically Pressed) at 930°C for 4h at a pressure of 150Mpa and its analysis is shown in table 1 but a limited amount of work was carried out on forged and heat treated Ti6Al4V. Cylindrical samples of

Ti6Al4V, which were large enough to prepare standard tensile and fatigue samples, were cut from these HIPped sample and some of these were taken to just above the transus and slowly cooled to room temperature. Some of these HIPped and slowly cooled samples were used to define the properties and microstructure in this standard condition and some of these samples were heat treated at 500°C for times up to 5 weeks in sealed tubes and analyses of the samples treated at 500°C are also shown in [table 1](#). Samples made from forged bar stock were also heat treated for 5 weeks at 500°C to assess the response of this starting material to this treatment. The samples were analysed after heat treatment to check on the extent of oxygen pick-up; the heat treated samples were assessed microstructurally and their tensile and fatigue properties compared with those of the initially slowly cooled samples.

Table 1. Analysis (wt%) of as- HIPped Ti6Al4V and after heat treating at 500°C for 5 weeks

Treatment	Ti	Al	V	Fe	Mo	O
As-HIPped	bal	6.51	3.89	0.18	-	0.20
500°C for 5 weeks	bal	6.48	3.87	0.18	-	0.20

Scanning electron microscopy was carried out at 20kV on a Philips XL30 to examine the fracture surfaces on samples tested in tension and in fatigue. Transmission electron microscopy was carried out on a JEOL 2100 operated at 200kV on the variously heat treated samples in order to assess whether alpha2 was detectable and on samples tested in tension and in fatigue to assess whether any alpha2, which may be present, influenced the dislocation behaviour. Atom probe work was carried out on an Imago LEAP 3000X HR on heat treated samples. The tensile tests were carried out at an initial strain rate of $1 \times 10^{-4} \text{s}^{-1}$ on a Zwick machine using samples with a diameter of 4mm and a gauge length of 20mm. The fatigue tests were carried out on an Amsler machine under load control at $R = 0.1$ (R is the ratio of the minimum load to the maximum load), using a frequency of 52Hz. The samples had a diameter of 3.6mm and gauge length of 20mm. Applied peak loads were selected in order to produce SN curves covering the range from 10^5 to 10^8 cycles. Samples which survived after more than 10^7 cycles are termed run-out. Phase equilibrium calculations were carried out using the Pandat software package, and the PanTitanium thermodynamic database developed by CompuTherm.

3. Results

3.1 Transmission electron microscopy.

(i) TEM of undeformed samples.

[Figure 1](#) shows a typical electron diffraction pattern taken from a sample of HIPped Ti6Al4V which was aged 5 weeks at 500°C and it is clear that the sample is ordered since obvious superlattice maxima are visible. A dark field micrograph, taken using one of the superlattice maxima visible in [figure 1a](#) is shown in [figure 1b](#) and it is clear that small particles of alpha 2 are distributed throughout the whole area. These are typically about 5nm in diameter but some are at least 10nm.

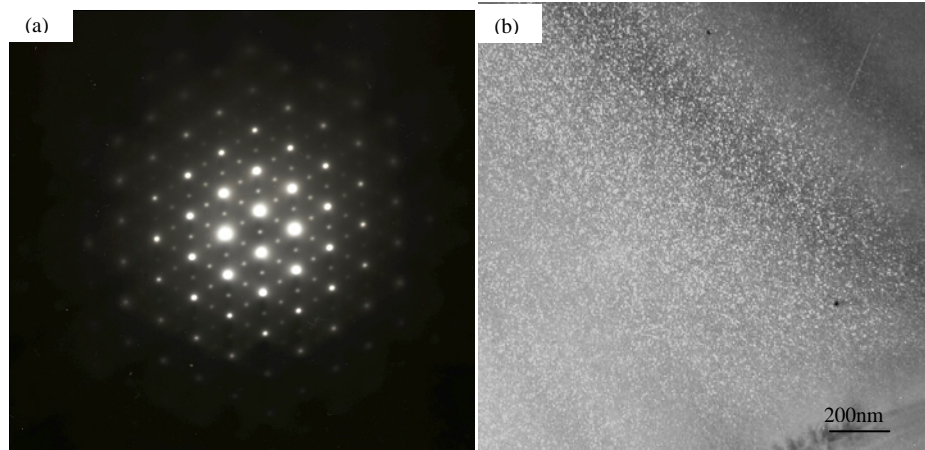


Figure 1 (a) Transmission electron diffraction pattern with the electron beam parallel to [0001] and (b) dark field micrograph taken using one of the superlattice maxima visible in (a) of Ti6Al4V aged at 500°C for 5 weeks.

Very weak superlattice maxima could be observed in slowly cooled samples by using Kikuchi lines from fundamental reflections to tilt to maximise the intensity of such a reflection, were it present and double exposing a diffraction pattern, initially with the objective aperture in the position to select the superlattice maximum, (although it cannot be seen on the screen) and then with the aperture removed. These maxima are just visible on suitably exposed negatives as shown in **figure 2(a)**; an intensity profile taken across this pattern is shown in **figure 2(b)** but it was not possible to obtain dark field images of domains from them because the signal to noise ratio was too small.

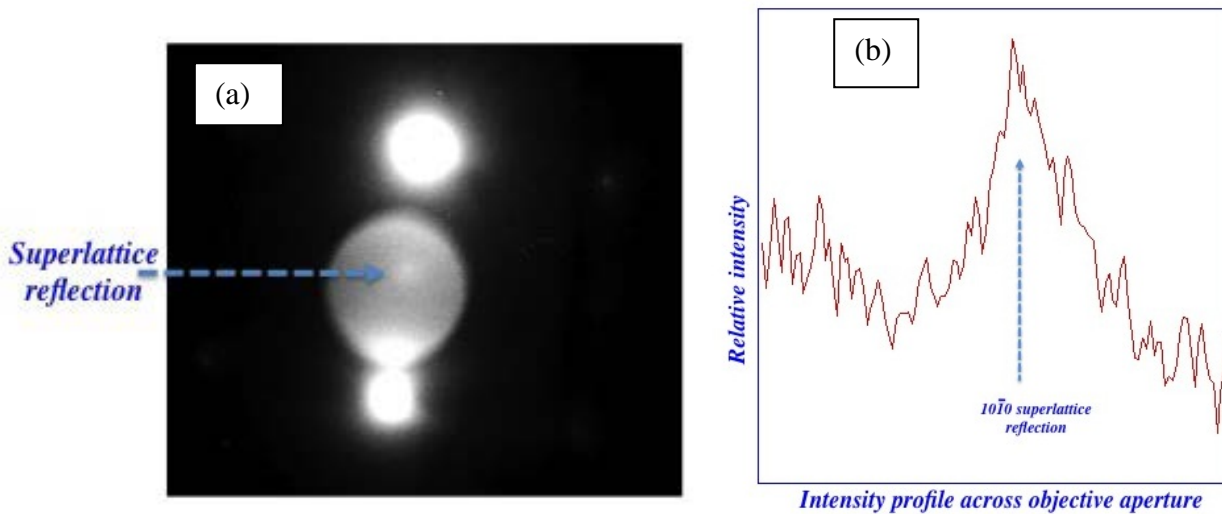


Figure 2. (a) Double exposed diffraction pattern taken from Ti6Al4V slowly cooled (5°C/min) from 930°C after orienting the sample to maximise the intensity of any 10-10 superlattice reflection present, with the electron beam direction close to [1-210]. (b) Intensity profile taken across the aperture through the weak superlattice maximum clearly showing the peak in intensity.

(ii) TEM of samples deformed in tension and fatigue

Typical images of a sample which had been heat treated at 500°C for 5 weeks and deformed in tension are shown in **figure 3**. In **figure 3(a)** planar slip is obvious and some weak fringes can be seen on the slip plane, whereas only irregular dislocations can be seen in (b), which was taken from the same sample. Very similar images could be obtained from slowly cooled samples, but planar slip was less obvious.

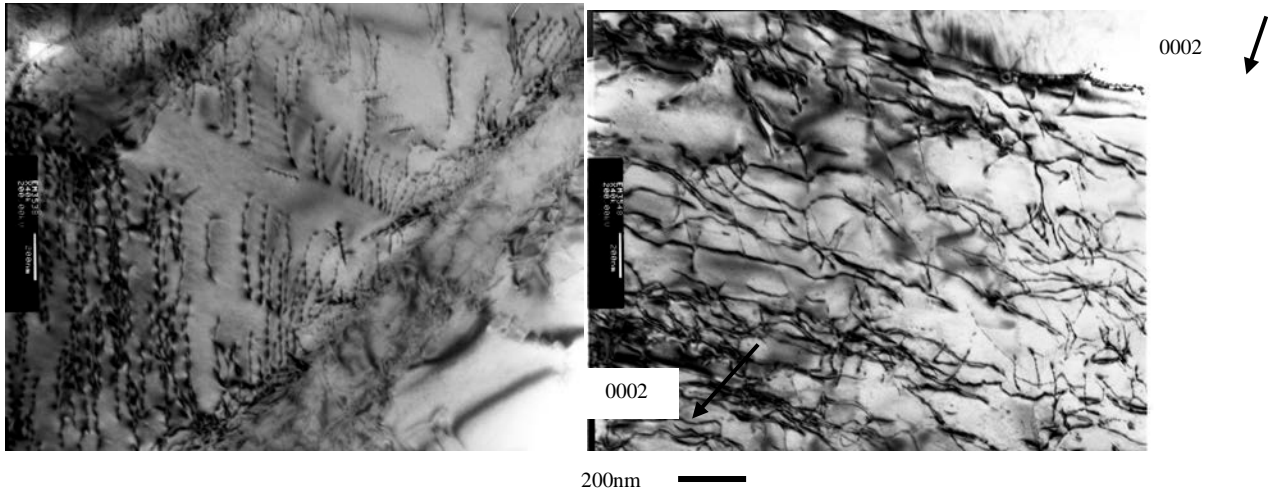


Figure 3. TEM images taken with $g = 0002$ with the electron beam direction close to $\langle 10\bar{1}0 \rangle$ of Ti64 which had been heat treated for 5 weeks at 500 °C and deformed to failure in tension showing in (a) obvious planar slip with associated weak fringes and in (b) more random c-component dislocations

A typical image of a slowly cooled sample deformed in fatigue is shown in [figure 4](#). Again it is clear that the slip is planar, but interestingly there is an indication of some pairing of dislocations (arrowed) of the same sign within the slip band.

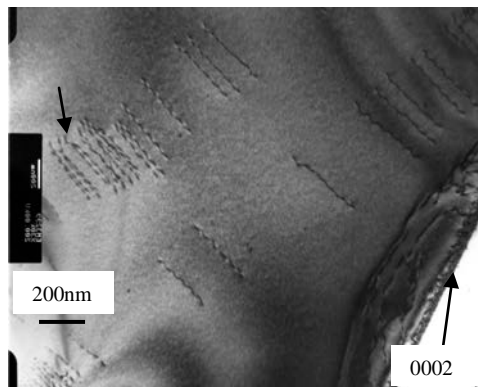


Figure 4 TEM image of c-component dislocations in a slowly cooled sample of Ti6Al4V fatigued to failure. Imaged with $g = 0002$ with B close to $\langle 10\bar{1}0 \rangle$. Dislocations pairs (arrowed) appear to be present.

A series of images taken from a sample which had been heat treated at 500 °C for 5 weeks before being fatigued to failure is shown in [figure 5](#). In this case it is apparent that dislocations are paired and because the images were taken using $\pm g$ pairs and the spacing did not change it is clear that they are pairs of like sign dislocations, as would be expected in an ordered alloy, where the first dislocation disorders the alloy and the second re-orders it. The fact that the spacing between pairs is not constant suggests on that basis that the extent of order varies from region to region, as would be inevitable since there are regions between domains that are not ordered and the domains are randomly distributed.

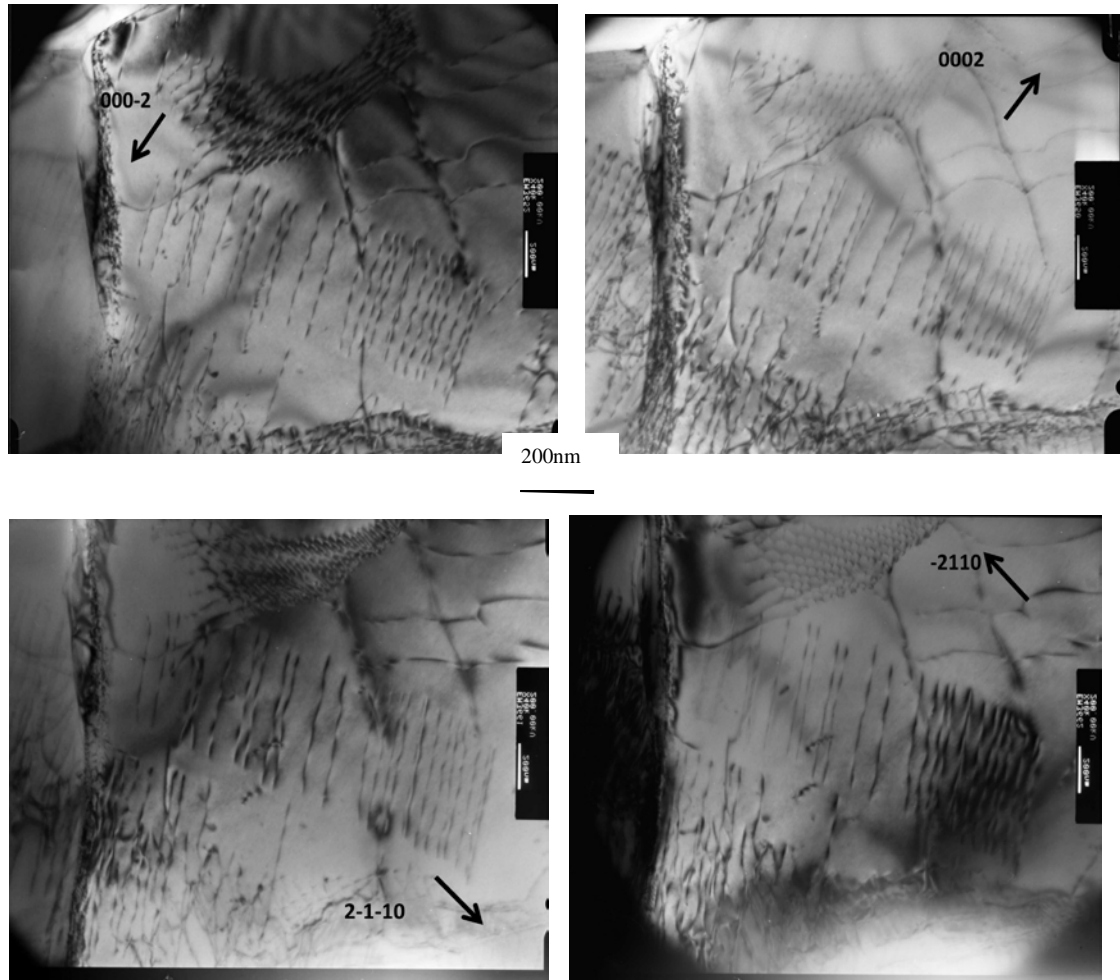


Figure 5 TEM images of dislocation pairs in Ti64 heat treated for 5 weeks at 500°C, fatigued to failure. Imaged with the diffracting vectors indicated with the beam direction close to $[0-110]$

(iii) Atom probe microscopy

Atom probe analysis was carried out of a sample which had been heat treated for 5 weeks at 500°C and the observations are illustrated in **figures 6 and 7**. These regions in figure 6 correspond to the alpha2 imaged in **figure 1** and show that the alpha2 has an Al concentration in the core of about 20at% but this drops to about 10at% on the outer region so that the alpha 2 is well below stoichiometry.

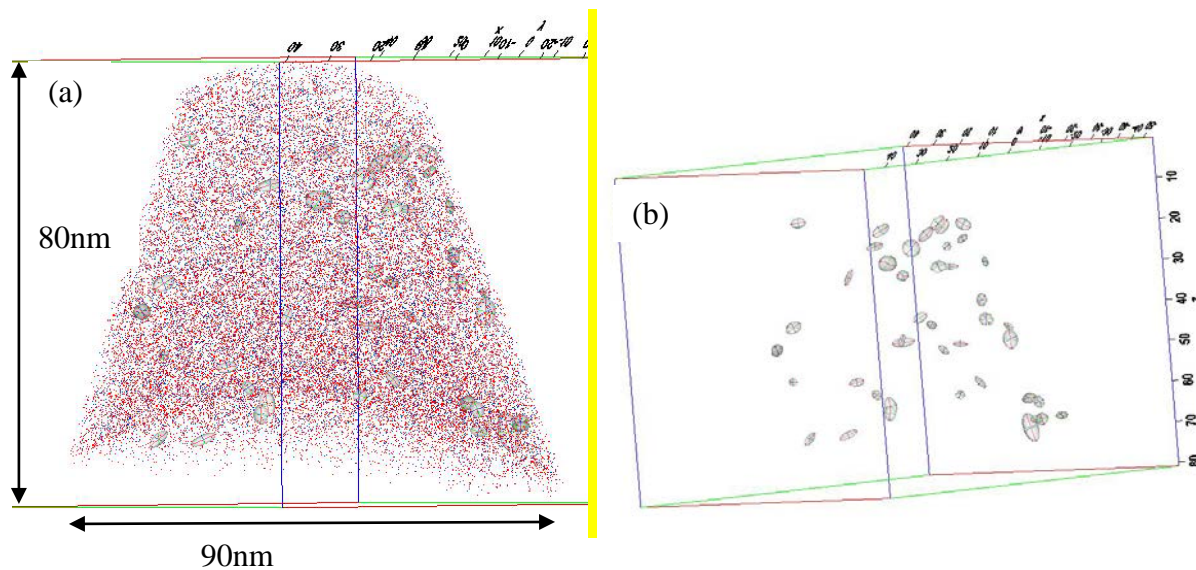


Fig 6 (a)Atom probe image of Ti6Al4V showing particles of Ti_3Al taken from a sample which had been heat treated at 500°C for 5 weeks.(b) Imge of the clusters

Many individual clusters were analysed and a typical example is shown in figure 7

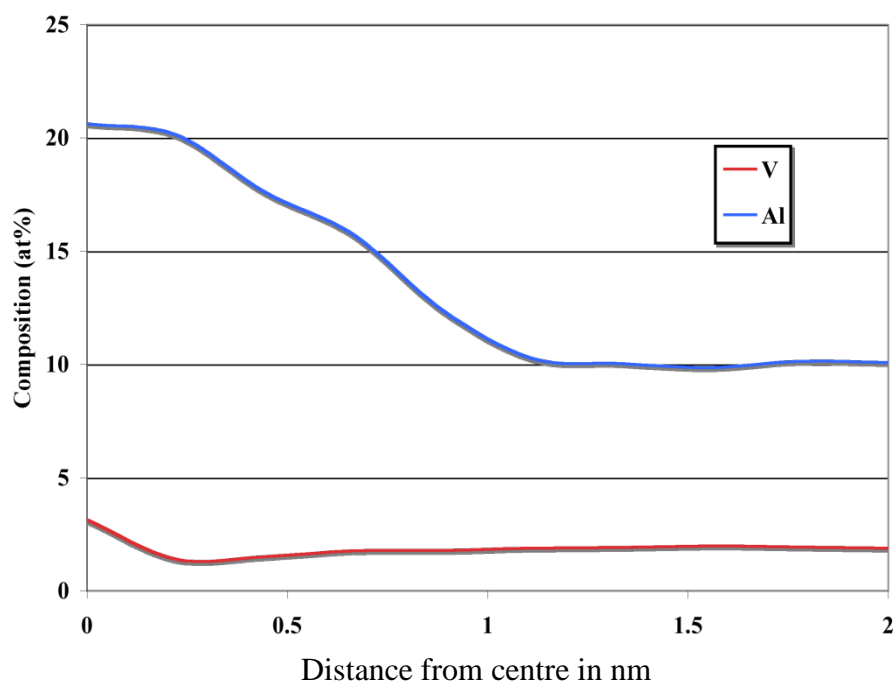


Figure 7 Analysis of an individual particle from a sample of Ti6Al4V analysed in the atom probe showing that the Al content varies from about 20at% at the centre to about 10at% in the outer region and there may also be a slight enrichment in V at the centre

3.2. Tensile properties of HIPped and of Heat-treated samples

Typical stress strain data for slowly cooled HIPped samples and for heat treated samples are shown in [figure 8](#) and there is a significant difference between the samples, with the heat treated sample having a

higher yield strength of over 1000MPa. The top three curves are from samples heat treated at 500°C for 5 weeks and the bottom three are from as-HIPped samples.

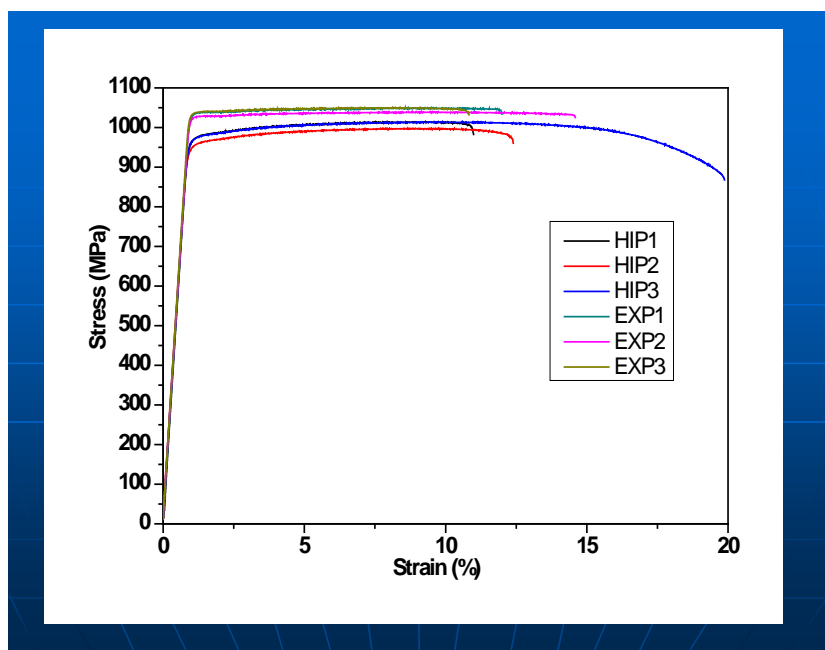


Figure 8 Tensile stress strain curves obtained for Ti6Al4V. The upper 3 curves are for the samples that were heat treated at 500C for 5 weeks and the lower 3 were in the as-HIPped condition.

It should be noted that analysis carried out on these samples (see table 1) shows no difference in the level of oxygen. Additional control experiments were also carried out on samples of forged and heat treated Ti64 and it was found that the tensile strength also increased after 5 weeks at 500°C but there was no reduction in ductility – it remained at about 20%, as shown in [table 2](#). Five samples were used for each condition and the scatter was very small. The lower tensile strength of the forged material (955Mpa) than the powder is presumably associated with slight differences in composition and the scale of the microstructures.

Table 2 Influence of heat treatment at 500°C for 5 weeks on forged Ti64

Process details	0.2% proof stress MPa	UTS MPa	% Elongation
Forged and heat treated at 925°C for 2h	878 +/- 4	926+/- 2	20+/- 1
Forged, heat treated at 925°C for 2h + 5weeks at 500°C	938+/-4	955+/- 3	21+/- 1

Detailed examination of the fracture surfaces (see below) of the HIPped samples shows that fracture sometimes initiated in regions where inclusions in the powder samples were present, but that the sample which failed after about 20% ductility showed a standard ductile failure. In view of the fact that with forged samples the strength increases on heat treating for 5 weeks at 500°C with no decrease in ductility it is inferred that the scatter between the samples shown in [figure 8](#) is not associated with the heat treatment, but is a problem with the powder cleanliness as shown below.

3.3 Fatigue properties of HIPped and of Heat-treated samples

The SN data obtained from the slowly cooled and aged samples are shown in **figure 9** and it appears that there is a large scatter in both groups of samples and it is not possible to determine from this limited data if there is any difference in fatigue properties.

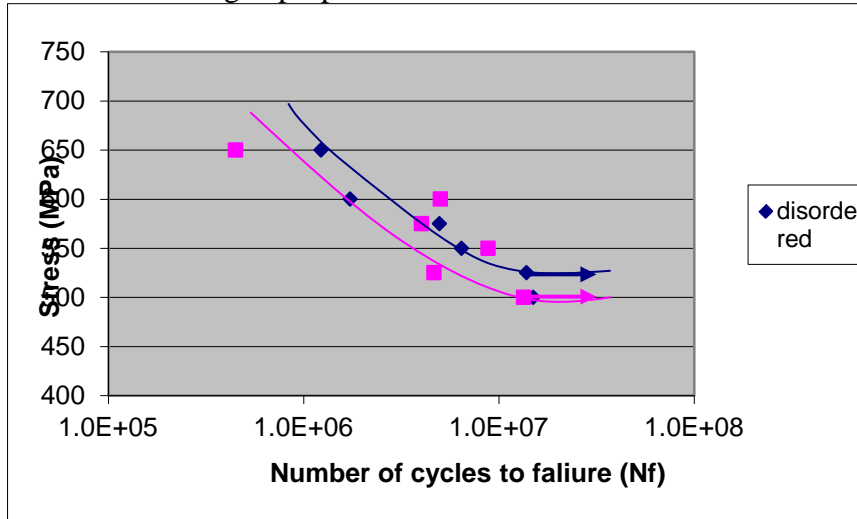


Figure 9 HCF SN data obtained from Ti6Al4V samples as-HIPped (◆) and from samples heat treated at 500°C for 5 weeks(.)

3.4. SEM observations of samples tested in tension and fatigue.

A micrograph of the fracture surface of a sample tested to failure in tension is shown in **figure 10**. Clearly the particular HIPped sample fractured in a ductile manner, but all other samples, whether they were as-HIPped or HIPped plus heat treated at 500°C for 5 weeks showed obvious fracture initiation sites associated with inclusions as illustrated in **figure 11**.

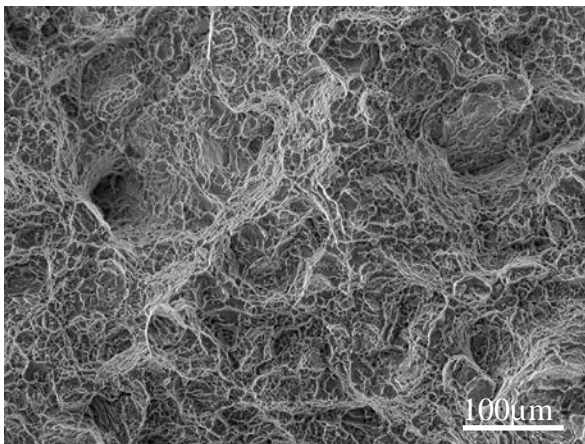


Figure 10. Secondary electron SEM image of fracture surface of Ti6Al4V as –HIPped which had a ductility of about 20%

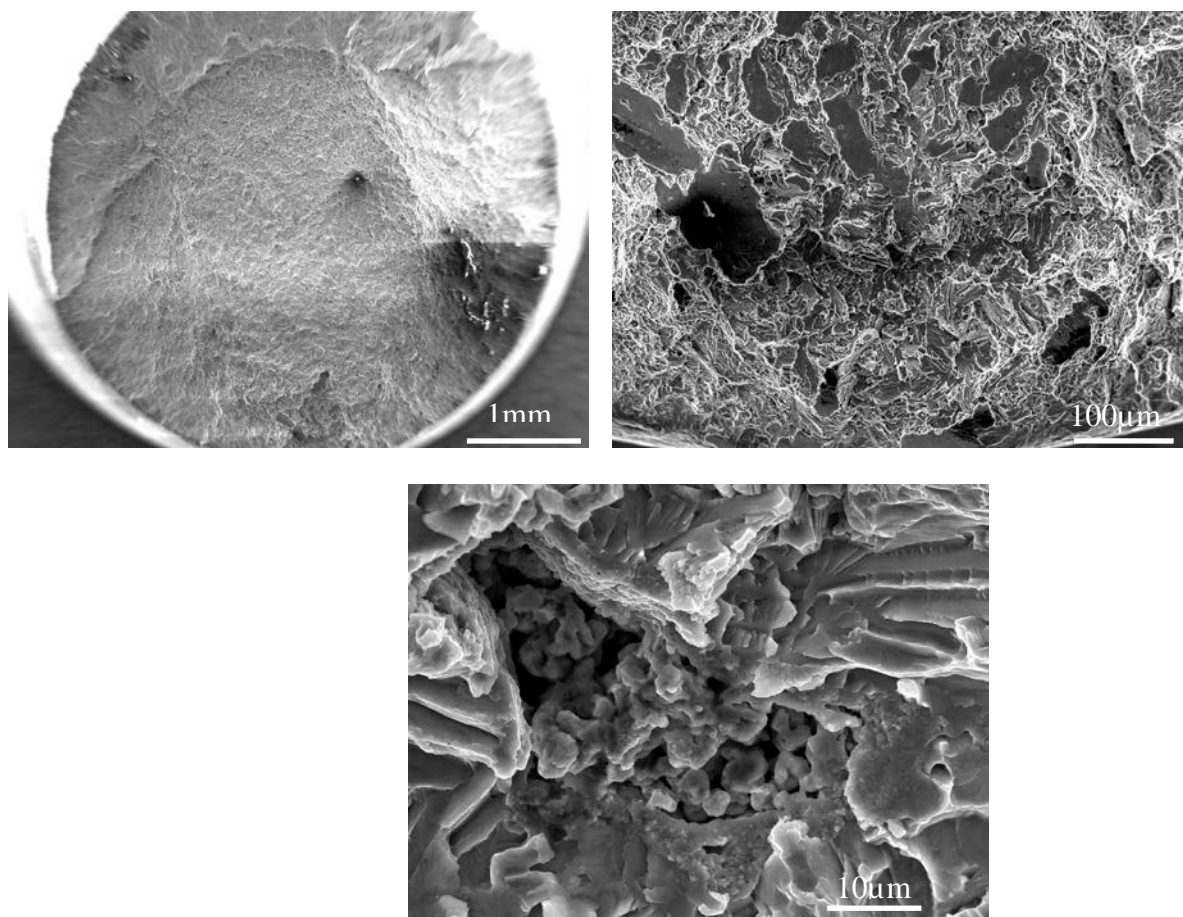


Figure 11 Secondary electron SEM images of fracture surface of as-HIPped Ti6Al4V which failed at 10% elongation.

4. Discussion

The formation of obvious domains of α_2 , which has been reported earlier [e.g 1] in suitably heat treated Ti64, has been confirmed in the present work. Indeed the presence of weak superlattice reflections in slowly cooled Ti64 is perhaps not surprising since α_2 is predicted to form below about 750°C [3].

The atom probe observations on the heat treated Ti64 have shown that the Al content is well below the predicted value of about 25% and presumably even longer heat treatments would be required to reach the stoichiometric composition.

The tensile strength of the ordered Ti6Al4V is significantly higher than that of the slowly cooled, HIPped samples and as noted earlier a similar increase of about 5% is found in forged samples which were heat treated at 500°C for 5 weeks and this increase in strength in forged samples is not associated with any decrease in ductility. This relatively small increase in strength associated with such a high density of small ordered domains is somewhat surprising, but since ordered domains are present in the slowly cooled samples, only a small increase may be expected because a comparison is being made between two samples with different extents of order rather than between an ordered and a disordered one. The scatter in ductility in the HIPped powder samples has been shown to be related to the presence of inclusions, although the minimum elongation observed was still above 10%. A similar scatter in the fatigue behaviour is probably

also associated with inclusions and either forged samples or powder samples prepared under more controlled conditions, would need to be used if the influence of heat treatment at 500°C on fatigue life were to be investigated. The higher strength observed in the samples tested in tension is presumably associated with the obvious influence of the microstructure on the behaviour of dislocations where paired dislocations are commonly seen in the samples heat treated at 500°C for 5 weeks. The images shown of paired dislocations were all obtained using $\mathbf{g} = 0002$ and the dislocations are clearly therefore of the type $\mathbf{b} = 1/3\langle 11-26 \rangle$ but other images have shown that dislocations with \mathbf{b} along $\langle 11-20 \rangle$ are also paired. The present work was done mainly with $\mathbf{g} = 0002$ close to a $\langle 10-10 \rangle$ beam direction so that it was straightforward to image the same area with a 1-210 type diffracting vector and thus ensure that all dislocations in the field of view would be in contrast and that any pairing or other unexpected shape was not influenced by out of contrast dislocations.

5. Conclusions

1 In Ti64 superlattice maxima are strongly visible after 5 weeks exposure at 500°C; and particles of alpha 2 can be imaged using these reflections. The maxima formed in slowly cooled samples are much weaker.

2 This ageing treatment increases the tensile strength.

3 Planar slip bands which contain paired dislocations are which are most obvious in fatigued samples which were heat treated for 5 weeks at 500°C are linked to the presence of the ordered domains of alpha2.

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